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TITLE: SEMIVOLATILE ORGANIC COMPOUNDS (GC/MS)
EPA Method 8270D

PREPARED BY:

Ismael Martínez Jiménez
Technical Representative

REVISED BY:

Karen Hernández Burgos
Team Leader Group

REVISED BY:

Heidy E. Alfonso Rivera
Operations Officer

APPROVED BY:

Iris M. Chévere Alfonzo
Laboratory Director

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Standard Operating Procedure for the Analyses of Semi-Volatile Organic Compounds (S-VOC's) by GC-Mass Spectrometry EPA Method 8270D

1.0 Scope and Application:

1.1 This method is used to measure the semi-volatile organic compounds in extracts prepared from aqueous matrices and/or solid matrices. The analyte list presented in this section identifies the possible group of compound amenable to this procedure; however, the analyte list presented in Appendix A is the group of analytes validated annually by the laboratory. This group of compounds is applicable to a variety of aqueous wastes matrices as well as aqueous sludge's, ground water, surface water, caustic liquors, acid liquors and water soluble solvents. The inclusion of additional analytes is limited only to performance evaluation of precision and accuracy studies using standards of known purity and successful completion of an MDL study.

1.2 It is the method of choice for TCLP extracts following any of the SW 846 extraction procedures.

1.3 It may also be applicable to Drinking water matrices but is not applicable for compliance purposes, the 500 series methods must be used instead.

1.4 Air samples can be tested when injecting with a gas tight syringe directly into the GCMS system.

1.5 The following analytes are listed in the method as applicable to be tested by this method; however, the sample preparative technique presented herein may not produce the adequate recovery needed for reliable results. Consult the method, the literature available and perform validations, identifying precision and accuracy as well as limit of detection studies before applying this procedure.

Compounds	CAS No. [a]
Acenaphthene	83-32-9
Acenaphthylene	208-96-8
Acetophenone	98-86-2
2-Acetylaminofluorene	53-96-3

Compounds	CAS No. [a]
1-Acetyl-2-thiourea	591-08-2
Aldrin	309-00-2
2-Aminoanthraquinone	117-79-3
Aminoazobenzene	60-09-3
4-Aminobiphenyl	92-67-1
3-Amino-9-ethylcarbazole	132-32-1
Anilazine	101-05-3
Aniline	62-53-3
o-Anisidine	90-04-0
Anthracene	120-12-7
Aramite	140-57-8
Aroclor 1016	12674-11-2
Aroclor 1221	11104-28-2
Aroclor 1232	11141-16-5
Aroclor 1242	53469-21-9
Aroclor 1248	12672-29-6
Aroclor 1254	11097-69-1
Aroclor 1260	11096-82-5
Azinphos-methyl	86-50-0
Barban	101-27-9
Benzidine	92-87-5
Benzoic acid	65-85-0
Benz(a)anthracene	56-55-3
Benzo(b)fluoranthene	205-99-2
Benzo(k)fluoranthene	207-08-9
Benzo(g,h,i)perylene	191-24-2
Benzo(a)pyrene	50-32-8
p-Benzoquinone	06-51-4
Benzyl alcohol	100-51-6
α-BHC	319-84-6
β-BHC	319-85-7
δ-BHC	319-86-8
γ-BHC (Lindane)	58-89-9
Bis(2-chloroethoxy)methane	111-91-1
Bis(2-chloroethyl) ether	111-44-4
Bis(2-chloroisopropyl) ether	39638-32-9
Bis(2-ethylhexyl) phthalate	117-81-7
4-Bromophenyl phenyl ether	101-55-3
Bromoxynil	1689-84-5
Butyl benzyl phthalate	85-68-7
Captafol	2425-06-1
Captan	133-06-2

Compounds	CAS No. [a]
Carbaryl	63-25-2
Carbofuran	1563-66-2
Carbophenothion	786-19-6
Chlordane (NOS)	57-74-9
Chlorfenvinphos	470-90-6
4-Chloroaniline	106-47-8
Chlorobenzilate	510-15-6
5-Chloro-2-methylaniline	95-79-4
4-Chloro-3-methylphenol	59-50-7
3-(Chloromethyl)pyridine hydrochloride	6959-48-4
1-Chloronaphthalene	90-13-1
2-Chloronaphthalene	91-58-7
2-Chlorophenol	95-57-8
4-Chloro-1,2-phenylenediamine	95-83-0
4-Chloro-1,3-phenylenediamine	5131-60-2
4-Chlorophenyl phenyl ether	7005-72-3
Chrysene	218-01-9
Coumaphos	56-72-4
p-Cresidine	120-71-8
Crotoxyphos	7700-17-6
2-Cyclohexyl-4,6-dinitro-phenol	131-89-5
4,4'-DDD	72-54-8
4,4'-DDE	72-55-9
4,4'-DDT	50-29-3
Demeton-O	298-03-3
Demeton-S	126-75-0
Diallate (*cis* or *trans*)	2303-16-4
2,4-Diaminotoluene	95-80-7
Dibenz(a,j)acridine	224-42-0
Dibenz(a,h)anthracene	53-70-3
Dibenzofuran	132-64-9
Dibenzo(a,e)pyrene	192-65-4
1,2-Dibromo-3-chloropropane	96-12-8
Di-n-butyl phthalate	84-74-2
Dichlone	117-80-6
1,2-Dichlorobenzene	95-50-1
1,3-Dichlorobenzene	541-73-1
1,4-Dichlorobenzene	106-46-7
3,3'-Dichlorobenzidine	91-94-1
2,4-Dichlorophenol	120-83-2

Compounds	CAS No. [a]
2,6-Dichlorophenol	87-65-0
Dichlorovos	62-73-7
Dicrotophos	141-66-2
Dieldrin	60-57-1
Diethyl phthalate	84-66-2
Diethylstilbestrol	56-53-1
Diethyl sulfate	64-67-5
Dimethoate	60-51-5
3,3'-Dimethoxybenzidine	119-90-4
Dimethylaminoazobenzene	60-11-7
7,12-Dimethylbenz(a)-anthracene	57-97-6
3,3'-Dimethylbenzidine	119-93-7
α,α-Dimethylphenethylamine	122-09-8
2,4-Dimethylphenol	105-67-9
Dimethyl phthalate	131-11-3
1,2-Dinitrobenzene	528-29-0
1,3-Dinitrobenzene	99-65-0
1,4-Dinitrobenzene	100-25-4
4,6-Dinitro-2-methylphenol	534-52-1
2,4-Dinitrophenol	51-28-5
2,4-Dinitrotoluene	121-14-2
2,6-Dinitrotoluene	606-20-2
Dinocap	39300-45-3
Dinoseb	88-85-7
Diphenylamine	122-39-4
5,5-Diphenylhydantoin	57-41-0
1,2-Diphenylhydrazine	122-66-7
Di-n-octyl phthalate	117-84-0
Disulfoton	298-04-4
Endosulfan I	959-98-8
Endosulfan II	33213-65-9
Endosulfan sulfate	1031-07-8
Endrin	72-20-8
Endrin aldehyde	7421-93-4
Endrin ketone	53494-70-5
EPN	2104-64-5
Ethion	563-12-2
Ethyl carbamate	51-79-6
Ethyl methanesulfonate	62-50-0
Famphur	52-85-7
Fensulfothion	115-90-2

Compounds	CAS No. [a]
Fenthion	55-38-9
Fluchloralin	33245-39-5
Fluoranthene	206-44-0
Fluorene	86-73-7
2-Fluorobiphenyl (surr)	321-60-8
2-Fluorophenol (surr)	367-12-4
Heptachlor	76-44-8
Heptachlor epoxide	1024-57-3
Hexachlorobenzene	118-74-1
Hexachlorobutadiene	87-68-3
Hexachlorocyclopentadiene	77-47-4
Hexachloroethane	67-72-1
Hexachlorophene	70-30-4
Hexachloropropene	1888-71-7
Hexamethylphosphoramide	680-31-9
Hydroquinone	123-31-9
Indeno(1,2,3-cd)pyrene	193-39-5
Isodrin	465-73-6
Isophorone	78-59-1
Isosafrole	120-58-1
Kepone	143-50-0
Leptophos	21609-90-5
Malathion	121-75-5
Maleic anhydride	108-31-6
Mestranol	72-33-3
Methapyrilene	91-80-5
Methoxychlor	72-43-5
3-Methylcholanthrene	56-49-5
4,4'-Methylenebis (2-chloroaniline)	101-14-4
4,4'-Methylenebis(*N*,*N*-dimethylaniline)	101-61-1
Methyl methanesulfonate	66-27-3
2-Methylnaphthalene	91-57-6
Methyl parathion	298-00-0
2-Methylphenol	95-48-7
3-Methylphenol	108-39-4
4-Methylphenol	106-44-5
Mevinphos	7786-34-7
Mexacarbate	315-18-4
Mirex	2385-85-5
Monocrotophos	6923-22-4
Naled	300-76-5

Compounds	CAS No. [a]
Naphthalene	91-20-3
1,4-Naphthoquinone	130-15-4
1-Naphthylamine	134-32-7
2-Naphthylamine	91-59-8
Nicotine	54-11-5
5-Nitroacenaphthene	602-87-9
2-Nitroaniline	88-74-4
3-Nitroaniline	99-09-2
4-Nitroaniline	100-01-6
5-Nitro-*o*-anisidine	99-59-2
Nitrobenzene	98-95-3
4-Nitrobiphenyl	92-93-3
Nitrofen	1836-75-5
2-Nitrophenol	88-75-5
4-Nitrophenol	100-02-7
5-Nitro-o-toluidine	99-55-8
Nitroquinoline-1-oxide	56-57-5
N-Nitrosodi-*n*-butylamine	924-16-3
N-Nitrosodiethylamine	55-18-5
N-Nitrosodimethylamine	62-75-9
N-Nitrosodiphenylamine	86-30-6
N-Nitrosodi-*n*-propylamine	621-64-7
N-Nitrosomethylethylamine	10595-95-6
N-Nitrosomorpholine	59-89-2
N-Nitrosopiperidine	100-75-4
N-Nitrosopyrrolidine	930-55-2
Octamethyl pyrophosphoramide	152-16-9
4,4'-Oxydianiline	101-80-4
Parathion	56-38-2
Pentachlorobenzene	608-93-5
Pentachloronitrobenzene	82-68-8
Pentachlorophenol	87-86-5
Phenacetin	62-44-2
Phenanthrene	85-01-8
Phenobarbital	50-06-6
Phenol	108-95-2
1,4-Phenylenediamine	106-50-3
Phorate	298-02-2
Phosalone	2310-17-0
Phosmet	732-11-6
Phosphamidon	13171-21-6
Phthalic anhydride	85-44-9

Compounds	CAS No. [a]
2-Picoline (2-Methylpyridine)	109-06-8
Piperonyl sulfoxide	120-62-7
Pronamide	23950-58-5
Propylthiouracil	51-52-5
Pyrene	129-00-0
Resorcinol	108-46-3
Safrole	94-59-7
Strychnine	57-24-9
Sulfallate	95-06-7
Terbufos	13071-79-9
1,2,4,5-Tetrachlorobenzene	95-94-3
2,3,4,6-Tetrachlorophenol	58-90-2
Tetrachlorvinphos	961-11-5
Tetraethyl dithiopyrophosphate	3689-24-5
Tetraethyl pyrophosphate	107-49-3
Thionazine	297-97-2
Thiophenol (Benzenethiol)	108-98-5
Toluene diisocyanate	584-84-9
o-Toluidine	95-53-4
Toxaphene	8001-35-2
1,2,4-Trichlorobenzene	120-82-1
2,4,5-Trichlorophenol	95-95-4
2,4,6-Trichlorophenol	88-06-2
Trifluralin	1582-09-8
2,4,5-Trimethylaniline	137-17-7
Trimethyl phosphate	512-56-1
1,3,5-Trinitrobenzene	99-35-4
Tris(2,3-dibromopropyl) phosphate	126-72-7
Tri-*p*-tolyl phosphate	78-32-0
O,O,O-Triethyl phosphorothioate	126-68-1

[a] Chemical Abstract Service Registry Number

2.0 Minimum Detection Limit (MDL):

2.1 The detection limits presented in Appendix A were performed during the last 12 months, they are statistical values based on the procedure found in 40 CFR part 136 Appendix B and were calculated from actual analyses on BEL's GC/MS instrument. The matrices were prepared in the laboratory with interference free reagents and materials. However, the practical quantitation limit (PQL) reported for each sample will depend on additional dilutions made if interferences or high values are encountered.

2.2 MDL determinations are performed annually on the most common matrices tested; however determinations on some of the least common matrices will be performed when the need is presented and samples become available.

3.0 Method Summary:

3.1 A measured volume of sample, approximately 1–L, is serially extracted with Methylene chloride at a pH greater than 11 and again at a pH less than 2 using a separatory funnel or a continuous extractor. The Methylene chloride extract is dried, concentrated to a volume of 1 mL, and analyzed by GC/MS.

3.2 The semi-volatile compounds tested through this method are introduced into the GC/MS system by direct injection of the dried concentrated solvent extraction. Qualitative identification of the parameters in the extract is performed using the retention time and the relative abundance of three characteristic masses (m/z). Quantitative analysis is performed using internal standard techniques with a single characteristic m/z.

3.3 The identification and quantification of the analytes is done through the software, however confirmation must be performed through visual inspection by the analyst. BEL assures adequate training, experience and supervision of the analyst, which is critical to acceptable performance in this test procedure.

3.4 The actual instrument conditions with gas flows, oven temperature ramp, injector and detector temperatures, valve timing, mass spectrophotometer voltages and the individual masses selected for detection, are all presented in Appendix B to this procedure.

4.0 Definitions:

The following definitions and acronyms are used in this method:

4.1 Initial Calibration Standards (ICS):
Standards prepared from pure analytes or commercially acquired in a mix for preparation of a calibration curve. A minimum of 5 standard concentrations levels are used to establish a working calibration range. The lowest of the group of standards should be at or below the necessary level to meet data quality objective of the project (or sample).

4.2 Initial Calibration Verification (ICV):
Standard prepared from pure analytes or commercially acquired in a mix for verification of the preparation of a calibration curve. It is of a different source or lot number (if from the same source) and analyzed at a mid-point of the calibration curve preferably with area

counts within 50-200% of the internal standards (IS). This verification is performed once after establishing the calibration curve, prior to its use.

4.3 Calibration Verification Standard (CVS):
A standard prepared from the same source of the calibration standards at mid level of the working range, used to verify the validity of the calibration curve by meeting established verification criteria. This verification should be performed once every 12 hours and prior to sample analyses.

4.4 Internal Standards (IS):
Standards introduced at the time of analyses that are used in the quantification formula for the analytes, they're retention times should be similar to that of the target analytes being tested and are prepared to a concentration of 40 ug/L. The IS compounds used are typically Deuterated to provide a means of GCMS differentiation from the target compounds with similar structures. They are added to all samples, standards and blanks and the system is considered in control if the area counts from consecutive runs agree within 50-200% for each IS.

4.4.1 The area counts for an acceptable mid-level concentration for a CVS should have all the target compounds within 50-200% of the IS area counts.

4.4.2 The internal standards selected in should permit most of the components of interest in a chromatogram to have retention times of 0.80 - 1.20, relative to one of the internal standards. Use the base peak ion from the specific internal standard as the primary ion for quantitation. If interferences are noted, use the next most intense ion as the quantitation ion.

4.5 Surrogate Standards (SS):
Standards introduced at the time of analyses that are used to monitor the systems performance during each run, it also monitors sample matrices and interference effects on the sample. The retention times should be similar to that of the target analytes being tested and are prepared to a concentration of 100 ug/L. The SS compounds used are typically Deuterated to provide a means of GCMS differentiation from the target compounds with similar structures. They are added to all samples, standards and blanks and the sample is considered in control if their concentration recovery is within a QC established range.

4.6 DFTPP Tuning:
DFTPP Tune is an MS calibration verification requirement that must be performed prior to commencing any calibration, verification or sample run and afterwards every 12 hours of run time. DFTPP criteria is established in the Chemstation software and in the method of reference, maintenance is required if the criteria can not be met. The standard may be

injected manually or be present in the standard mix (at the prescribed concentration) for subsequent evaluation and acceptance of sample batches.

4.7 Matrix Spike (MS):
A sample fortified with target compounds is an MS, the recoveries calculated after subtracting analytes present in the sample are used to evaluate accuracy of the test procedure in a particular matrix. Duplicate matrix spikes (MSD) performed on the same sample are used to evaluate precision statistics for that particular matrix.

4.8 Retention Time (RT):
Time of elution of a peak from the moment of injection to entrance and being detected by the Mass Spectrometer is called the absolute retention time (RT). The relative retention time is the elution time relative to another compound, in this case the IS peak established during the compound list compilation in the Chemstation software (see Appendix B).

4.9 A Laboratory Control Sample (LCS) should be included with each analytical batch. The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. The LCS is spiked with the same analytes at the same concentrations as the matrix spike. When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix.

5.0 Interferences and Contamination:

5.1 Interferences from Helium Gases, reagent water, reagents, standards, sorbent traps, non-PTFE lines tubing and rubber equipment material will be detected by the instrument. Phthalate esters are common contaminants that leach from plasticizers in rubber or plastic materials used in the sample preparation. They produce false positives that will be typically observed in the blank analyses run, subtraction of these false positive from the samples is not permitted. However the situation should cause the sample result to be flagged and a narrative explaining the finding presented in the report.

5.2 The following compounds may require special treatment when being determined by this method:

- 5.2.1 Benzidine may be subject to oxidative losses during solvent concentration and its chromatographic behavior is poor.
- 5.2.2 Under the alkaline conditions of the extraction step from aqueous matrices, α-BHC, γ-BHC, Endosulfan I and II, and Endrin are subject to decomposition. Neutral extraction should be performed if these compounds are expected to be present.

5.2.3 Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, chemical reaction in acetone solution, and photochemical decomposition.

5.2.4 N-nitrosodimethylamine is difficult to separate from the solvent under the chromatographic conditions described.

5.2.5 N-nitrosodiphenylamine decomposes in the gas chromatographic inlet and cannot be separated from diphenylamine. For this reason, it is acceptable to report the combined result for n-nitrosodiphenylamine and diphenylamine for either of these compounds as a combined concentration.

5.2.6 1,2-Diphenylhydrazine is unstable even at room temperature and readily converts to azobenzene. Given the stability problems, it would be acceptable to calibrate for 1,2-diphenylhydrazine using azobenzene. Under these poor compound separation circumstances the results for either of these compounds should be reported as a combined concentration.

5.2.7 Pentachlorophenol, 2,4-dinitrophenol, 4-nitrophenol, benzoic acid, 4,6-dinitro-2-methylphenol, 4-chloro-3-methylphenol, 2-nitroaniline, 3-nitroaniline, 4-nitroaniline, and benzyl alcohol are subject to erratic chromatographic behavior, especially if the GC system is contaminated with high boiling material.

5.2.8 Pyridine may perform poorly at the GC injection port temperatures listed in this method. Lowering the injection port temperature may reduce the amount of degradation. However, the analyst must use caution in modifying the injection port temperature, as the performance of other analytes may be adversely affected. Therefore, if pyridine is to be determined in addition to other target analytes, it may be necessary to perform separate analyses. In addition, pyridine may be lost during the evaporative concentration of the sample extract. As a result, this may yield low recoveries unless great care is exercised during the concentration steps.

6.0 Safety:

6.1 The analyst should be aware that the Methylene Chloride used in the extraction by this method is a suspected carcinogen as is the majority of the compounds tested by this method, their standards should be handled with protective clothing in a well ventilated hood.

6.2 The instrument consumes Helium; this gas is typically supplied in cylinders. Proper gas cylinder handling techniques, gas lines connection and cylinder support are crucial to avoid accidents. An improperly supported cylinder that tips over and breaks the regulator or valve can become a dangerous gas propulsion object.

7.0 Equipment and Supplies:

7.1 **Agilent Model 7683 automatic liquid sampler** for sample introduction, once the device injects the sample into the GC column it sends the start signal to the GC, which will subsequently begin data acquisition and temperature programming.

7.2 **Agilent Injection port liners** designed for liquid sample introduction. It should be inserted 1 cm into the liner. Liners are the primary collection surface for contamination when injecting liquid directly into the system.

7.3 **Agilent Model 6890N, Gas Chromatographic system** complete with a Chemstation data collection & data management software, temperature programmable oven, electronic pressure control, valve timing for gas flow movement, sample introduction interface and split-less injection port. It should also have the capacity to maintain constant flow through the column during desorption and temperature program cycle.

7.4 **Agilent Chemstation Data system** - A computer based system that allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program is interfaced to the mass spectrometer. The computer software allows searching any GC/MS data file for ions of a specified mass and plotting such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software allows integrating the abundances in any EICP between specified time or scan-number limits. The recent version of the EPA/NIST Mass Spectral Library is also integrated.

7.5 **Agilent GC/MS interface** – The interface is a direct coupling device, connection is accomplished by inserting the column directly into the mass spectrometer, it is used for 30 m x 0.32 mm x 0.25 um ID columns.

8.0 Reagents and Standards:

8.1 Commercially prepared standard mixes containing the target compounds typically tested are used by BEL in this method. The mix may contain more analytes than needed for the target list or it may be custom purchased containing only the targets. It is important to obtain the same standards list from two alternate sources or from the same source but two different lot numbers because verification checks are performed with second source standards. A second source is considered a separate vendor or a different lot number from the same vendor.

8.2 Internal Standards and Surrogate Standards (SS) are purchased separately from the target compounds. No second sources are used here.

8.3 Methylene Chloride used to dilute commercial standards and used in extractions must be of the highest quality, always purchase a brand that states that it is suitable for extraction analyses containing the lowest residue after evaporation. Pesticide grade is also a suitable grade.

8.4 A water purification system (Millipore Super-Q or equivalent) may be used to generate reagent water.

9.0 Sample Collection, Preservation, Shipment and Storage:

9.1 Samples are collected in pre-cleaned new glass 1 liter bottles with Teflon lined covers. The sample is typically a composite sample and at least 1 liter bottle.

9.2 If the sample has residual chlorine it should be removed with ascorbic acid, the use of sodium thiosulfate is discouraged because sulfates interfere in the mass spectrometer.

9.3 The samples must be shipped in ice and/or stored in a cooler at below 4°C; the analyst has 7 days to perform the extraction. Once extracted the concentrate can be stored in a sealed vial for 40 days at <4 °C in the refrigerator prior to analyses.

10.0 Quality Control:

10.1 Quality Control procedures are necessary to evaluate the GC system operation as well as its stability throughout the run. The sample matrix amenability to the process is also tested with matrix spikes and matrix spike duplicates.

10.2 The GC/MS system must be tuned to meet the DFTPP specifications in Sections 11.3.1 and 11.4.1of EPA method 8270D.

10.3 There must be an initial calibration of the GC/MS system as described in following Section 11.0 of this procedure. In addition, the initial calibration curve should be verified immediately after performing the standard analyses using a second source standard (prepared using standards different from the calibration standards). The suggested acceptance limits for this initial calibration verification analysis are 70 - 130%.

10.4 The GC/MS system must meet the DFTPP specification criteria described in Table 3 of the 8270D method, each 12 hours.

10.5 Initial Demonstration of Proficiency (also known as Initial Demonstration of Capability) - Each analyst must demonstrate initial proficiency with each sample preparation and determinative method combination it utilizes, by generating data of acceptable accuracy and precision for target analytes in a clean matrix. The analyst must also repeat this initial demonstration whenever significant changes in instrumentation are made. This is accomplished by analyzing 4 fortified samples, of the matrix under evaluation, and calculating the average % recovery and the relative standard deviation between the four samples. They should produce similar recovery and precision data as produced by the laboratory in the past. The samples should be fortified with the target analytes at 10 to 50 times the established laboratory method detection limit (MDL).

10.5.1 When the samples are fortified at 10 times the laboratory desired MDL, the same data can be used to establish this MDL, only 3 additional samples must be prepared and run to complete the 7 needed for an MDL study. The criteria established in 40 CFR parts 136, appendix B; still apply and must be meet for the data to be used to establish an MDL.

10.6 Before processing any samples, the analyst should demonstrate, through the analyses of a method blank that interferences from the analytical system, glassware, and reagents are under control. Each time a set of samples is analyzed or there is a change in reagents, a method blank should be analyzed as a safeguard against chronic laboratory contamination. The blanks should be carried through all stages of sample preparation and measurement.

10.7 A Laboratory Control Sample (LCS) should be included with each analytical batch. The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. The LCS is spiked with the same analytes at the same concentrations as the matrix spike. When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix.

10.8 Documenting the effect of the matrix should include the analysis of at least one matrix spike and one duplicate unspiked sample or one matrix spike/matrix spike duplicate pair. The decision on whether to prepare and analyze duplicate samples or a matrix spike/matrix spike duplicate must be based on knowledge of the samples in the sample batch. If samples are expected to contain target analytes, then laboratories may use one matrix spike and a duplicate analysis of an unspiked field sample. If samples are not expected to contain target analytes, laboratories should use a matrix spike and matrix spike duplicate pair.

10.9 Surrogate recoveries - The analyst must evaluate surrogate recovery data from

individual samples versus the surrogate control limits developed by the laboratory in previous analyses. Samples with surrogate recoveries under 10% must be re-injected; if the poor recovery persists the result must be reported with a flag noting the occurrence. The LCS recovery is very useful in these situations demonstrating that the system can perform acceptably in a clean matrix.

10.10 The experience of the analyst performing GC/MS analyses is invaluable to the success of the methods. Each day that analysis is performed, the calibration verification standard should be evaluated to determine if the chromatographic system is operating properly. Questions that should be asked are:

Do the peaks look normal?
Is the response obtained comparable to the response from previous calibrations?
Careful examination of the standard chromatogram can indicate whether the column is still performing acceptably, the injector is leaking, the injector septum needs replacing, etc.
If any changes are made to the system (e.g., the column changed), recalibration of the system must take place.

11.0 Calibration and Standardization:

11.1 Mass spectrometer – The Mass Spectrometer is instructed, through the software, to scan from 35 to 500 amu every 2 sec or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. Prior to use the mass spectrometer must be adjusted (tuned) to produce a mass spectrum for DFTPP which meets all of the criteria in Table 3 (found in 8270 D method) when 2 uL of 25 ug/mL of the GC/MS tuning standard DFTPP are injected through the GC. To ensure sufficient precision of mass spectral data, the software is set for a scan rate of five spectra samplings, to allow for a desirable MS acquisition of a sample component that elutes from the GC.

11.2 Once injected and run the software, upon request, will evaluate the spectra of DFTPP to assure compliance with the criteria of Table 3 in the method. Bring the spectra to the screen from the data file and click on DFTPP evaluation, it is best to use the auto-find feature when you evaluate DFTPP because the feature automatically performs as the method requires. This feature will also produce a hard copy of the DFTPP evaluation for your records. No calibration or other use of the system can be performed until a passing spectrum is produced. The DFTPP method conditions can be shortened if only DFTPP is tested or until the last peak elutes if DFTPP is mixed with other performance testing analytes. DFTPP can be placed in the auto sampler for injections on long sequences. It is best to perform and pass the first DFTPP test before leaving subsequent tests in the sampler. The DFTPP tuning verification must be performed every 12 hours of testing.

11.3 The GC/MS tuning standard solution should also be used to assess GC column performance and injection port inertness. Degradation of DDT to DDE and DDD should not exceed 20%. (The Chemstation software will perform this calculation from the menu) Benzidine and pentachlorophenol should be present at their normal responses, and should not exceed a tailing factor of 2 given by the following equation:

Tailing Factor = BC/AB

Where the peak is defined as follows: AC is the width at 10% height; DE is the height of peak and B is the height at 10% of DE. This equation compares the width of the back half of the peak to the width of the front half of the peak at 10% of the height. The Chemstation software will perform this calculation from the menu.

11.4 If degradation is excessive and/or poor chromatography is noted, the injection port may require cleaning. It may also be necessary to break off the first 6 to12 in. of the capillary column. The use of a guard column between the injection port and the analytical column will help prolong analytical column performance life.

11.5 Proceed with the analysis of the calibration standards, inject 1-2 uL. Tabulate the area response of the characteristic ions against the concentration for each target analyte and each internal standard. Calculate response factors (RF) for each target analyte relative to one of the internal standards. The internal standard selected for the calculation of the RF for a target analyte should be the internal standard that has a retention time closest to the analyte being measured.

The RF is calculated as follows:

$$RF = \frac{As \times Cis}{Ais \times Cs}$$

Where:
A s = Peak area (or height) of the analyte or surrogate.
A is = Peak area (or height) of the internal standard.
C s = Concentration of the analyte or surrogate.
C is = Concentration of the internal standard.

11.6 Following the calibration curve preparation and analyses, the Chemstation software will calculate a mean Response Factor (RF) for each target compound. Upon request the software will produce a table with the mean response factor and the relative standard deviation (RSD) of the response factors for each target analyte. The RSD should be less than or equal to 20% for each target analyte. It is also recommended that a minimum response factor for the most common target analytes be reached, as noted in Table 4 (of the 8270D method), and be demonstrated for each individual calibration level as a means to ensure that these compounds are behaving as expected. This

information is written into the developed method and is evaluated upon request to the software (see entire printed method in appendix B) In addition, meeting the minimum response factor criteria for the lowest calibration standard is critical in establishing and demonstrating the desired sensitivity.

11.6.1 If more than 10% of the compounds included with the initial calibration exceed the 20% RSD limit and alternatively, do not meet the minimum correlation coefficient (0.99) for alternate curve fits, then the chromatographic system is considered too reactive for analysis to begin. Clean or replace the injector liner and/or capillary column, then repeat the calibration procedure.

11.7 Linearity of target analytes, If the RSD of any target analyte is 20% or less, then the response factor is assumed to be constant over the calibration range, and the average response factor may be used for quantitation. If the RSD of any target analyte is greater than 20%, refer to Method 8000 for additional calibration options. One of the options must be applied to GC/MS calibration in this situation, or a new initial calibration must be performed.

11.7.1 When the RSD exceeds 20%, the plotting and visual inspection of a calibration curve can be a useful diagnostic tool. The inspection may indicate analytical problems, including errors in standard preparation, the presence of active sites in the chromatographic system, analytes that exhibit poor chromatographic behavior, etc. The software also plots the calibration curve for this visual inspection.

11.8 GC/MS calibration verification; the verification consists of three steps that are performed at the beginning of each 12-hour analytical shift.

11.8.1 Prior to the analysis of samples or calibration standards, inject or introduce 50 ng of DFTPP into the GC/MS system. The resultant mass spectra for DFTPP must meet the criteria given in Table 3 (of the 8270D method) before sample analysis begins. These criteria must be demonstrated each 12-hour shift during which samples are analyzed.

11.8.2 The initial calibration curve for each compound of interest should be verified prior to sample analysis, using a second source mid-point standard. The result should agree within 30% of the expected concentration. From their on, the calibration curve must be verified once every 12 hrs. This is accomplished by analyzing a calibration standard at a concentration near the midpoint concentration for the calibrating range of the GC/MS. the calibration verification standard should meet the minimum response factors as noted in Table 4 (of the 8270D method), this criteria is also written into the developed method and easily verified through the software.

NOTE: The DFTPP and calibration verification standard may be combined into a single standard as long as both tuning and calibration verification acceptance criteria for the project can be met without interferences.

11.8.3 All target compounds of interest must be evaluated using a 20% criterion. Use percent difference when performing the average response factor model calibration. Use percent drift when calibrating using a regression fit model. This criterion is also written into the developed method and easily verified through the software.

11.8.4 A method blank should be analyzed after the calibration standard, or at any other time during the analytical shift, to ensure that the total system (introduction device, transfer lines and GC/MS system) is free of contaminants. If the method blank indicates contamination, then it may be appropriate to analyze a solvent blank to demonstrate that the contamination is not a result of carryover from standards or samples.

11.9 Internal standard retention time; the retention times of the internal standards in the calibration verification standard must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 seconds from the time established in the mid-point standard level of the most recent initial calibration sequence, then the chromatographic system must be inspected for malfunctions and corrections must be made, as required. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

11.10 Internal standard response; if the Extracted Ion Current Profile (EICP) area for any of the internal standards in the calibration verification standard changes by a factor of two (-50% to + 100%) from that in the mid-point standard level of the most recent initial calibration sequence, the mass spectrometer must be inspected for malfunctions and corrections must be made, as appropriate. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

12.0 Procedure:

12.1 The extraction of the sample must follow one of the extraction methods (3510C for aqueous samples, 3540C or 3550C for solid/soil samples). All sample extracts and standard solutions must be allowed to warm to ambient temperature before analysis. Add surrogates at time of sample extraction.

12.2 For aqueous samples, they are usually extracted using separatory funnel techniques. If emulsions cannot be broken with a glass rod or by adding Sodium Chloride (like Morton Salt) and it is clear that this will prevent achieving acceptable solvent recovery with separatory funnel extractions, continuous extraction may be used. The

separatory funnel extraction scheme described herein assumes a sample volume of 1 L. When sample volumes of 2 L are to be extracted, use 250, 100, and 100-mL volumes of Methylene chloride for the serial extraction of the base/neutrals and 200, 100, and 100-mL volumes of Methylene chloride for the acids.

12.3 Mark the water meniscus on the side of the sample bottle for later determination of sample volume. Pour the entire sample into a 2–L separatory funnel. Pipette 1.00 mL of the surrogate standard spiking solution into the separatory funnel and mix well. Check the pH of the sample with wide-range pH paper and adjust to pH>11 with Sodium Hydroxide solution.

12.4 Add 60 mL of Methylene chloride to the sample bottle, seal, and shake for 30 s to rinse the inner surface. Transfer the solvent to the separatory funnel and extract the sample by shaking the funnel for 2 min. with periodic venting to release excess pressure. Allow the organic layer to separate from the water phase for a minimum of 10 min. If the emulsion interface between layers is more than one-third the volume of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample, but may include stirring, filtration of the emulsion through glass wool, centrifugation, or other physical methods. Collect the Methylene chloride extract in a 250 mL Erlenmeyer flask. If the emulsion cannot be broken (recovery of less than 80% of the Methylene chloride, corrected for the water solubility of Methylene chloride), transfer the sample, solvent, and emulsion into the extraction chamber of a continuous extractor and proceed to extract as described in Section 11.3 of method 625.

12.5 Add a second 60-mL volume of Methylene chloride to the sample bottle and repeat the extraction procedure a second time, combining the extracts in the Erlenmeyer flask. Perform a third extraction in the same manner. Label the combined extract as the base/neutral fraction.

12.6 Adjust the pH of the aqueous phase to less than 2 using sulfuric acid. Serially extract the acidified aqueous phase three times with 60-mL aliquots of Methylene chloride. Collect and combine the extracts in a 250-mL Erlenmeyer flask and label the combined extracts as the acid fraction.

12.7 If you are performing Base/Neutral and Acid analysis the total contents of the extractions can be combined and evaporated down, if not then for each fraction, pour the contents in a 1 mL Turbo-Vap evaporation flask. Bring the volume down while verifiying at least twice the volume and during this verification, rinse the walls of the flask with Methylene chloride. Bring the volume down below 1 mL without letting the solvent dry in the flask.

12.8 Quantitatively transfer the contents to a 1 mL volumetric flask and bring to volume with the solvent, cap and mix. Transfer the contents to an auto sampler vial.

12.9 Determine the original sample volume by refilling the sample bottle to the mark and transferring the liquid to a 1000 mL graduated cylinder. Record the sample volume to the nearest 5 mL. Note this volume for final calculations.

12.10 Just prior to sample analyses add 10 uL of the Internal Standards to all samples, standards and blanks, mix.

12.11 If the response for any quantitation ion exceeds the initial calibration range of the GC/MS system, the sample extract must be diluted and reanalyzed. Additional internal standard solution must be added to the diluted extract to maintain the same concentration as in the calibration standards.

12.12 If the initial analysis of the sample or a dilution of the sample has a concentration of any analyte that exceeds the initial calibration range, the sample must be reanalyzed at a higher dilution. Secondary ion quantitation is allowed only when there are sample interferences with the primary ion.

12.13 When ions from a compound in the sample saturate the detector, this analysis must be followed by the analysis of organic-free reagent water blank. If the blank analysis is not free of interferences, then the system must be decontaminated. Sample analysis may not resume until the blank analysis is demonstrated to be free of interferences.

13.0 Calculation:

13.1 The calculations are performed by the Chemstation software, if a dilution was made and the appropriate multiplication factor applied in the software, no further calculations need be done. If the dilution was performed and not entered into the data system then a manual calculation is required. If other than 1 liter of sample was extracted then apply the following equation:

Conc. Ug/L = 1000/ sample volume (mls)

13.2 Qualitative identification is performed by observing the relative peak heights of the three characteristic masses in the EICPs, chosen in the software for each analyte, must fall within ±20% of the relative intensities of these masses in a reference mass spectrum. The reference mass spectrum can be obtained from a standard analyzed in the GC/MS system or from the Wiley/NIST reference library.

14.0 Method Performance:

14.1 Method performance is annually calculated from the QA/QC data reported for the samples, this information can be found in Annual QA/QC Report for semivolatiles.

14.2 As a quality control check of method performance, the laboratory spikes all samples with the surrogate standard spiking solutions, and calculates the percent recovery (R) of each surrogate compound. As part of the QC program for the laboratory, method accuracy for wastewater samples are assessed and records are maintained. From the analysis of spiked wastewater samples, calculate the average percent recovery and the standard deviation of the percent recovery (s_R). Express the accuracy assessment as a percent recovery interval from % recovery $-2s_R$ to % recovery$+2$ s_R. For example, if % recovery =90% and s_R =10%, the accuracy interval is expressed as 70-110%. Update of the accuracy assessment for each parameter is performed on an annual basis.

14.3 Method performance is also evaluated through Performance Evaluation (PE) Studies submitted to the laboratory by third party providers approved by EPA.

15.0 Pollution Prevention and Waste Management:

15.1 The laboratory pollution prevention and waste management program teaches the analyst where to dispose of his/her waste by department. This waste is segregated in labeled satellite areas throughout the laboratory prior to movement to the storage area for removal by a transporting and disposal entity in Lab-PACs.

15.2 Manifest are prepared and kept on file for inspection.

16.0 Data Assessment and Acceptance Criteria for Quality Control Measures:

16.1 The laboratory QA/QC department assesses data produced by each department following supervisor approval.

16.2 Acceptance criteria are established for all methods through data produced in the method. When insufficient data is available the method acceptance or performance criteria is used, in all cases the more stringent of the two are implemented.

17.0 Corrective Action for Out-of-Control Data:

17.1 BEL's QA/QC system is designed to monitor and detect Out-of-Control data as soon as it occurs. When the occurrence is detected the system is placed out of service until the appropriate corrective action can be administered.

17.2 Instruments are serviced by manufacturer trained professionals; BEL also has in-house professionals experienced in preventive maintenance techniques to diagnose problems that will enhance the preparation of a service technician prior to his arrival.

18.0 Contingencies for Out-of-Control Data or Unacceptable Data:

18.1 Out-of-Control data is retested if sufficient sample is available and/or the holding time has not expired. Otherwise data produced under these situations is flagged and a narrative is submitted with the report.

19.0 References:

19.1 SW846 Method 8000B

19.2 SW 846 Method 8270D

19.3 SW 846 Method 8000 update letter of December 17, 2007

APPENDIX A
Analyte list with Laboratory Method Detection Limit

Beckton Environmental Laboratories
Method Detection Limit 2010-2011

Area: Gas Chromatography
Analysis: **Semi - Volatiles**
Method: **TCLP 8270C**

Instrument: GC-MSD 6890N/5973N

Analyst: Amelia Echevarria
Date:7-16-22 April 2010
Expiration date:April 2011
Concentration:10 ppm
Matrix: Liquid

Contaminant	MDL ppm
Pyridine	0.002
2-Methylphenol	0.002
3&4-Methylphenol	0.006
Hexachloroethane	0.004
Nitrobenzene	0.003
Hexachlorobutadiene	0.004
2,4,6-Trichlorophenol	0.004
2,4,5-Trichlorophenol	0.003
2,4-Dinitrotoluene	0.003
Hexachlorobenzene	0.002
Pentachlorophenol	0.001

APPENDIX B
Entire Method Printout from HP Chemstation Software

TOPLEVEL PARAMETERS

Method Information For: C:\MSDCHEM\1\METHODS\DFTPP.M

Method Sections To Run:

(X) Save Copy of Method With Data
( ) Pre-Run Cmd/Macro =
(X) Data Acquisition
(X) Data Analysis
( ) Post-Run Cmd/Macro =

Method Comments:
Hydrocarbon Fingerprinting By ASTM D 5729-06SBL-5ms Column

END OF TOPLEVEL PARAMETERS

INSTRUMENT CONTROL PARAMETERS

Sample Inlet: GC
Injection Source: GC ALS
Mass Spectrometer: Enabled

6890 GC METHOD

COLUMN

Initial temp: 125 'C (On)
Initial time: 0.00 min
Ramps:

#	Rate	Final temp	Final time
1	15.00	270	0.00
2	0.0(Off)		

Post temp: 0 'C
Post time: 0.00 min
Run time: 9.67 min

Maximum temp: 360 'C
Equilibration time: 0.10 min

FRONT INLET (UNKNOWN)
Mode: Splitless
Initial temp: 250 'C (On)
Pressure: 11.91 psi (On)
Purge flow: 50.1 mL/min
Purge time: 0.75 min
Total flow: 54.1 mL/min
Gas saver: On
Saver flow: 20.0 mL/min
Saver time: 0.75 min
Gas type: Helium

BACK INLET ()

COLUMN 1
Capillary Column
Model Number: Agilent 19091S-433
HP-5MS, 0.25mm * 30m * 0.25um
Max temperature: 350 'C
Nominal length: 30.0 m
Nominal diameter: 250.00 um
Nominal film thickness: 0.25 um
Mode: constant flow
Initial flow: 1.0 mL/min
Nominal init pressure: 11.92 psi

COLUMN 2
(not installed)

Average velocity: 38 cm/sec
Inlet: Front Inlet
Outlet: MSD
Outlet pressure: vacuum

FRONT DETECTOR (NO DET)

SIGNAL 1
Data rate: 20 Hz
Type: test plot
Save Data: Off
Zero: 0.0 (Off)
Range: 0
Fast Peaks: Off
Attenuation: 0

BACK DETECTOR (NO DET)

SIGNAL 2
Data rate: 20 Hz
Type: test plot
Save Data: Off
Zero: 0.0 (Off)
Range: 0
Fast Peaks: Off
Attenuation: 0

COLUMN COMP 1
(No Detectors Installed)

COLUMN COMP 2
(No Detectors Installed)

THERMAL AUX 2
Use: MSD Transfer Line Heater
Description: Transfer Line
Initial temp: 280 'C (On)
Initial time: 0.00 min

#	Rate	Final temp	Final time
1	0.0	(Off)	

POST RUN
Post Time: 0.00 min

TIME TABLE

Time	Specifier	Parameter & Setpoint

7673 Injector

Front Injector:

Sample Washes	1	
Sample Pumps	2	
Injection Volume	1.0	microliters
Syringe Size	10.0	microliters
PostInj Solvent A Washes	2	
PostInj Solvent B Washes	2	
Viscosity Delay	1	seconds
Plunger Speed	Fast	
PreInjection Dwell	0.00	minutes
PostInjection Dwell	0.00	minutes

Back Injector:
No parameters specified

MS ACQUISITION PARAMETERS

General Information
------- -----------

Tune File : atune.u
Acquistion Mode : Scan

MS Information
-- -----------

Solvent Delay : 3.40 min

EM Absolute : True
Resulting EM Voltage : 2000.0

[Scan Parameters]

Low Mass : 35.0

High Mass : 500.0
Threshold : 500
Sample # : 2 A/D Samples 4
Plot 2 low mass : 50.0
Plot 2 high mass : 550.0

[MSZones]

MS Quad : 150 C maximum 200 C
MS Source : 250 C maximum 250 C

END OF MS ACQUISITION PARAMETERS

END OF INSTRUMENT CONTROL PARAMETERS

DATA ANALYSIS PARAMETERS

Method Name: C:\MSDCHEM\1\METHODS\DFTPP.M

Percent Report Settings

Sort By: Signal

Output Destination
Screen: Yes
Printer: No
File: No

Integration Events: Meth Default

Generate Report During Run Method: No

Signal Correlation Window: 0.020

Quantitative Report Settings

Report Type: Summary

Output Destination
Screen: Yes
Printer: No
File: No

Generate Report During Run Method: No

Semi-volatiles Methods 625/8270C
Calibration Last Updated: Wed Jun 09 15:53:17 2010

erence Window: 2.00 Minutes
Non-Reference Window: 1.00 Minutes
Correlation Window: 0.10 minutes
Default Multiplier: 1.00
Default Sample Concentration: 0.00

Compound Information

---

1) Penthachlorophenol ( )

Ret. Time 5.17 min., Extract & Integrate from 5.13 to 5.29 min.

Lvl ID Conc (ug/mL) Response

Curve Fit: Avg. RF

---

2) Benzidine ( )

Ret. Time 7.55 min., Extract & Integrate from 7.50 to 7.64 min.

Lvl ID Conc (ug/mL) Response

Curve Fit: Avg. RF

---

3) 4,4-DDT ( )

Ret. Time 8.39 min., Extract & Integrate from 8.36 to 8.44 min.

Lvl ID Conc (ug/mL) Response

Curve Fit: Avg. RF

END OF DATA ANALYSIS PARAMETERS

Thu Jun 10 15:22:06 2010

Additional Information for TCLP.M
File created Thu Feb 14 11:39:51 2002

Method C:\MSDCHEM\1\METHODS\TCLP.M renamed as C:\MSDCHEM\1\METHODS\METHANOL.M
Tue Apr 15 11:10:48 2003

Method C:\MSDCHEM\1\METHODS\TCLP.M renamed as C:\MSDCHEM\1\METHODS\TCLP1.M
Wed Dec 17 15:28:06 2003

Method C:\MSDCHEM\1\METHODS\TCLP1.M renamed as C:\MSDCHEM\1\METHODS\TCLP2.M
Wed Dec 17 15:30:00 2003

Method C:\MSDCHEM\1\METHODS\TCLP1.M renamed as C:\MSDCHEM\1\METHODS\TCLP2.M
Tue Sep 28 11:31:37 2004

Method C:\MSDCHEM\1\METHODS\TCLP2.M renamed as C:\MSDCHEM\1\METHODS\TCLP.M
Thu Jan 20 08:50:49 2005

Method C:\MSDCHEM\1\METHODS\TCLP.M renamed as C:\MSDCHEM\1\METHODS\TCLP2.M
Wed Mar 02 08:53:59 2005

Method C:\MSDCHEM\1\METHODS\TCLP.M renamed as C:\MSDCHEM\1\METHODS\TCLP1.M
Fri Apr 01 08:41:19 2005

Method C:\MSDCHEM\1\METHODS\TCLP.M renamed as C:\MSDCHEM\1\METHODS\TCLP1.M
Mon Jul 11 14:53:10 2005

Method C:\MSDCHEM\1\METHODS\TCLP.M renamed as C:\MSDCHEM\1\METHODS\TCLP2.M
Sun Oct 02 13:40:43 2005

Method C:\MSDCHEM\1\METHODS\TCLP.M renamed as C:\MSDCHEM\1\METHODS\TCLP2.M
Tue Jan 24 09:59:42 2006

Method C:\MSDCHEM\1\METHODS\TCLP2.M renamed as C:\MSDCHEM\1\METHODS\TCLP.M
Sat Mar 04 09:28:42 2006

Method C:\MSDCHEM\1\METHODS\TCLP.M renamed as C:\MSDCHEM\1\METHODS\CRES-4,6PHE.M
Wed Mar 07 14:30:11 2007

Method C:\MSDCHEM\1\METHODS\TCLP.M renamed as C:\MSDCHEM\1\METHODS\TCLP1.M
Fri Aug 31 09:45:41 2007

Method C:\MSDCHEM\1\METHODS\TCLP.M renamed as C:\MSDCHEM\1\METHODS\TEST.M
Thu Oct 04 10:18:54 2007

Method C:\MSDCHEM\1\METHODS\TCLP.M renamed as C:\MSDCHEM\1\METHODS\DFTPP.M
Fri Oct 05 09:07:10 2007

TOPLEVEL PARAMETERS

Method Information For: C:\MSDCHEM\1\METHODS\TCLP-7.M

Method Sections To Run:

(X) Save Copy of Method With Data
( ) Pre-Run Cmd/Macro =
(X) Data Acquisition
(X) Data Analysis
( ) Post-Run Cmd/Macro =

Method Comments:
This is the TCLP8270 Semi-Volatiles method.

END OF TOPLEVEL PARAMETERS

INSTRUMENT CONTROL PARAMETERS

Sample Inlet: GC
Injection Source: GC ALS
Mass Spectrometer: Enabled

6890 GC METHOD

OVEN
Initial temp: 40 'C (On)
Initial time: 2.00 min
Ramps:

#	Rate	Final temp	Final time
1	22.00	240	0.00
2	10.00	330	0.00
3	0.0	(Off)	

Post temp: 0 'C
Post time: 0.00 min
Run time: 20.09 min
Maximum temp: 360 'C
Equilibration time: 0.00 min

FRONT INLET (UNKNOWN)
Mode: Splitless
Initial temp: 250 'C (On)
Pressure: 6.89 psi (On)
Purge flow: 50.0 mL/min
Purge time: 0.75 min
Total flow: 54.1 mL/min
Gas saver: On
Saver flow: 20.0 mL/min
Saver time: 0.75 min
Gas type: Helium

BACK INLET ()

COLUMN 1
Capillary Column
Model Number: Agilent 19091S-433
HP-5MS, 0.25mm * 30m * 0.25um
Max temperature: 350 'C
Nominal length: 30.0 m
Nominal diameter: 250.00 um
Nominal film thickness: 0.25 um
Mode: constant flow
Initial flow: 1.0 mL/min

COLUMN 2
(not installed)

Nominal init pressure: 6.89 psi
Average velocity: 36 cm/sec
Inlet: Front Inlet
Outlet: MSD
Outlet pressure: vacuum

FRONT DETECTOR (NO DET)

SIGNAL 1
Data rate: 20 Hz
Type: test plot
Save Data: Off
Zero: 0.0 (Off)
Range: 0
Fast Peaks: Off
Attenuation: 0

COLUMN COMP 1
(No Detectors Installed)

BACK DETECTOR (NO DET)

SIGNAL 2
Data rate: 20 Hz
Type: test plot
Save Data: Off
Zero: 0.0 (Off)
Range: 0
Fast Peaks: Off
Attenuation: 0

COLUMN COMP 2
(No Detectors Installed)

THERMAL AUX 2
Use: MSD Transfer Line Heater
Description: Transfer Line
Initial temp: 330 'C (On)
Initial time: 0.00 min

#	Rate	Final temp	Final time
1	0.0	(Off)	

POST RUN
Post Time: 0.00 min

TIME TABLE

Time	Specifier	Parameter & Setpoint

7673 Injector

Front Injector:

Sample Washes	1	
Sample Pumps	2	
Injection Volume	2.0	microliters
Syringe Size	10.0	microliters
PostInj Solvent A Washes	2	
PostInj Solvent B Washes	2	
Viscosity Delay	1	seconds
Plunger Speed	Fast	
PreInjection Dwell	0.00	minutes
PostInjection Dwell	0.00	minutes

Back Injector:
No parameters specified

MS ACQUISITION PARAMETERS

General Information
------- -----------

Tune File : ATUNE.U
Acquistion Mode : Scan

MS Information
-- -----------

Solvent Delay : 2.27 min

Absolute : True
Resulting EM Voltage : 2000.0

[Scan Parameters]

Low Mass : 50.0
High Mass : 550.0
Threshold : 500
Sample # : 2 A/D Samples 4
Plot 2 low mass : 50.0
Plot 2 high mass : 550.0

[Zones]

MS Quad : 150 C maximum 200 C
MS Source : 250 C maximum 250 C

END OF MS ACQUISITION PARAMETERS

END OF INSTRUMENT CONTROL PARAMETERS

DATA ANALYSIS PARAMETERS

Method Name: C:\MSDCHEM\1\METHODS\TCLP-7.M

Percent Report Settings

Sort By: Signal

Output Destination
Screen: Yes
Printer: No
File: No

Integration Events: AutoIntegrate

Generate Report During Run Method: No

Signal Correlation Window: 0.020

Quantitative Report Settings

Report Type: Summary

Output Destination
Screen: Yes
Printer: No
File: No

Generate Report During Run Method: No

SEMIVOLATILES OC 8270C-TCLP
Calibration Last Updated: Fri Sep 11 09:38:53 2009

Reference Window: 10.00 Minutes
Non-Reference Window: 5.00 Minutes
Correlation Window: 0.02 minutes
Default Multiplier: 1.00

Default Sample Concentration: 0.00

Compound Information

1) 1,4-DiCHLOROBENZENE-d4 (ISTD TR)
Ret. Time 5.28 min., Extract & Integrate from 5.21 to 5.39 min.

Lvl	ID	Conc (ug/ml)	Response
10		40.000	18835747
20		40.000	21332546
30		40.000	20638741
50		40.000	23563105
60		40.000	22717389
80		40.000	21255907

ISTD conc: 40.000 ug/ml
Curve Fit: Avg. RF

2) PYRIDINE ( )
Ret. Time 2.63 min., Extract & Integrate from 2.61 to 3.05 min.

Lvl	ID	Conc (ug/ml)	Response
10		10.000	1036674
20		20.000	3020591
30		30.000	5234163
50		50.000	8070791
60		60.000	7764958
80		80.000	12391093

Curve Fit: Avg. RF

3) 2-FLUOROPHENOL ( )
Ret. Time 3.95 min., Extract & Integrate from 3.84 to 4.14 min.

Lvl	ID	Conc (ug/ml)	Response
10		100.000	29365399
20		100.000	29023403
30		100.000	30556803
50		100.000	29184506
60		100.000	27361376
80		100.000	28564815

Curve Fit: Avg. RF

4) PHENOL-d5 ( )
Ret. Time 4.98 min., Extract & Integrate from 4.85 to 5.11 min.

Lvl	ID	Conc (ug/ml)	Response
10		100.000	25424445
20		100.000	24571723
30		100.000	26495935
50		100.000	25062430
60		100.000	24065393
80		100.000	25125625

Curve Fit: Avg. RF

5) 2-METHYLPHENOL ( )
Ret. Time 5.64 min., Extract & Integrate from 5.55 to 5.74 min.

Lvl	ID	Conc (ug/ml)	Response
10		10.000	4705228
20		20.000	8847718

30	30.000	15003064
50	50.000	24255915
60	60.000	25639933
80	80.000	-1

Curve Fit: Avg. RF

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6) 3&4-METHYLPHENOL ( )

Ret. Time 5.88 min., Extract & Integrate from 5.77 to 5.93 min.

Lvl	ID	Conc (ug/ml)	Response
10		20.000	3238698
20		40.000	6386758
30		60.000	6909456
50		100.000	15604166
60		120.000	17806766
80		160.000	23426691

Curve Fit: Avg. RF

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7) NAPHTHALENE-d8 (ISTD TR)

Ret. Time 6.64 min., Extract & Integrate from 6.52 to 6.68 min.

Lvl	ID	Conc (ug/ml)	Response
10		40.000	30887852
20		40.000	32975612
30		40.000	31438991
50		40.000	33146817
60		40.000	31458845
80		40.000	30570729

ISTD conc: 40.000 ug/ml

Curve Fit: Avg. RF

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8) HEXACHLOROETHANE ( )

Ret. Time 5.81 min., Extract & Integrate from 5.77 to 5.89 min.

Lvl	ID	Conc (ug/ml)	Response
10		10.000	3247769
20		20.000	5504574
30		30.000	11278493
50		50.000	17078965
60		60.000	18293214
80		80.000	-1

Curve Fit: Avg. RF

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9) NITROBENZENE-d5 ( )

Ret. Time 5.87 min., Extract & Integrate from 5.80 to 5.94 min.

Lvl	ID	Conc (ug/ml)	Response
10		100.000	25965779
20		100.000	25864967
30		100.000	27622161
50		100.000	24664455
60		100.000	21069825
80		100.000	15630556

Curve Fit: Avg. RF

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10) NITROBENZENE ( )

Ret. Time 5.93 min., Extract & Integrate from 5.87 to 6.01 min.

Lvl	ID	Conc (ug/ml)	Response
10		10.000	2834186
20		20.000	7041425
30		30.000	9867510
50		50.000	16303973
60		60.000	17303574
80		80.000	-1

Curve Fit: Avg. RF

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11) HEXACHLOROBUTADIENE ( )
Ret. Time 6.81 min., Extract & Integrate from 6.74 to 6.86 min.

Lvl	ID	Conc (ug/ml)	Response
10		10.000	3047238
20		20.000	5585449
30		30.000	11822328
50		50.000	13095158
60		60.000	18116192
80		80.000	20112231

Curve Fit: Avg. RF

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12) ACENAPHTHENE-d10 (ISTD TR)
Ret. Time 8.56 min., Extract & Integrate from 8.45 to 8.73 min.

Lvl	ID	Conc (ug/ml)	Response
10		40.000	22022041
20		40.000	23813358
30		40.000	22837564
50		40.000	24672501
60		40.000	23417735
80		40.000	22354190

ISTD conc: 40.000 ug/ml
Curve Fit: Avg. RF

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13) 2,4,6-TRICHLOROPHENOL ( )
Ret. Time 7.77 min., Extract & Integrate from 7.69 to 7.83 min.

Lvl	ID	Conc (ug/ml)	Response
10		10.000	1957676
20		20.000	4809125
30		30.000	7113413
50		50.000	11450963
60		60.000	13490448
80		80.000	14107835

Curve Fit: Avg. RF

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14) 2,4,5-TRICHLOROPHENOL ( )
Ret. Time 7.77 min., Extract & Integrate from 7.77 to 7.94 min.

Lvl	ID	Conc (ug/ml)	Response
10		10.000	1560736
20		20.000	3850512
30		30.000	5634770
50		50.000	8114827
60		60.000	8480929
80		80.000	9466474

Curve Fit: Avg. RF

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15) 2-FLUOROBIPHENYL ( )
Ret. Time 7.84 min., Extract & Integrate from 7.78 to 7.93 min.

Lvl	ID	Conc (ug/ml)	Response
10		100.000	52974404
20		100.000	53087501
30		100.000	54674513
5[redacted]		100.000	52341212
6[redacted]		100.000	50726608
80		100.000	52385827

Curve Fit: Avg. RF

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16) 2,4-DINITROTOLUENE ( )
Ret. Time 8.83 min., Extract & Integrate from 8.75 to 8.90 min.

Lvl	ID	Conc (ug/ml)	Response
10		10.000	3100380
20		20.000	7066310
30		30.000	10140964
50		50.000	17651760
60		60.000	20561434
80		80.000	21716314

Curve Fit: Avg. RF

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17) 2,4,6-TRIBROMOPHENOL ( )
Ret. Time 9.45 min., Extract & Integrate from 9.36 to 9.58 min.

Lvl	ID	Conc (ug/ml)	Response
10		100.000	12583221
20		100.000	12520885
30		100.000	13180215
5[redacted]		100.000	12535491
6[redacted]		100.000	12055441
80		100.000	12554902

Curve Fit: Avg. RF

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18) PHENANTHRENE-d10 (ISTD TR)
Ret. Time 10.18 min., Extract & Integrate from 10.10 to 10.31 min.

Lvl	ID	Conc (ug/ml)	Response
10		40.000	31621373
20		40.000	34041509
30		40.000	32010203
50		40.000	34356259
60		40.000	32197594
80		40.000	31038362

ISTD conc: 40.000 ug/ml
Curve Fit: Avg. RF

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19) HEXACHLOROBENZENE ( )
Ret. Time 9.78 min., Extract & Integrate from 9.58 to 9.98 min.

Lvl	ID	Conc (ug/ml)	Response
10		10.000	4225663
20		20.000	8060985
30		30.000	10307630
5[redacted]		50.000	19633806
6[redacted]		60.000	21106269
80		80.000	24205187

Curve Fit: Avg. RF

20) PENTACHLOROPHENOL ( )
Ret. Time 10.04 min., Extract & Integrate from 9.98 to 10.15 min.

Lvl	ID	Conc (ug/ml)	Response
[redacted]		10.000	1856891
[redacted]		20.000	3176017
30		30.000	4685629
50		50.000	8850483
60		60.000	12052704
80		80.000	15902391

Curve Fit: Avg. RF

21) CHRYSENE-d12 (ISTD TR)
Ret. Time 13.61 min., Extract & Integrate from 13.41 to 13.81 min.

Lvl	ID	Conc (ug/ml)	Response
10		40.000	39688067
20		40.000	43295308
30		40.000	39404611
50		40.000	42637145
60		40.000	38466464
80		40.000	39108857

ISTD conc: 40.000 ug/ml
Curve Fit: Avg. RF

22) TERPHENYL-d14 ( )
Ret. Time 12.08 min., Extract & Integrate from 11.88 to 12.28 min.

Lvl	ID	Conc (ug/ml)	Response
[redacted]		100.000	67506012
[redacted]		100.000	65558258
30		100.000	69744065
50		100.000	65173431
60		100.000	63955410
80		100.000	65079740

Curve Fit: Avg. RF

23) PERYLENE-d12 (ISTD TR)
Ret. Time 16.06 min., Extract & Integrate from 15.86 to 16.26 min.

Lvl	ID	Conc (ug/ml)	Response
10		40.000	35967279
20		40.000	41720444
30		40.000	38098343
50		40.000	40019775
60		40.000	35984037
80		40.000	35813374

ISTD conc: 40.000 ug/ml
Curve Fit: Avg. RF

END OF DATA ANALYSIS PARAMETERS

Thu Jun 10 15:18:54 2010

Additional Information for TCLP.M
File created Thu Feb 14 11:39:51 2002

Method C:\MSDCHEM\1\METHODS\TCLP.M renamed as C:\MSDCHEM\1\METHODS\METHANOL.M
Tue Apr 15 11:10:48 2003

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[redacted]d Dec 17 15:28:06 2003

Method C:\MSDCHEM\1\METHODS\TCLP1.M renamed as C:\MSDCHEM\1\METHODS\TCLP2.M
Wed Dec 17 15:30:00 2003

Method C:\MSDCHEM\1\METHODS\TCLP1.M renamed as C:\MSDCHEM\1\METHODS\TCLP2.M
Tue Sep 28 11:31:37 2004

Method C:\MSDCHEM\1\METHODS\TCLP2.M renamed as C:\MSDCHEM\1\METHODS\TCLP.M
Thu Jan 20 08:50:49 2005

Method C:\MSDCHEM\1\METHODS\TCLP.M renamed as C:\MSDCHEM\1\METHODS\TCLP2.M
Wed Mar 02 08:53:59 2005

Method C:\MSDCHEM\1\METHODS\TCLP.M renamed as C:\MSDCHEM\1\METHODS\TCLP1.M
Fri Apr 01 08:41:19 2005

Method C:\MSDCHEM\1\METHODS\TCLP.M renamed as C:\MSDCHEM\1\METHODS\TCLP1.M
Mon Jul 11 14:53:10 2005

Method C:\MSDCHEM\1\METHODS\TCLP.M renamed as C:\MSDCHEM\1\METHODS\TCLP2.M
Sun Oct 02 13:40:43 2005
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Method C:\MSDCHEM\1\METHODS\TCLP.M renamed as C:\MSDCHEM\1\METHODS\TCLP2.M
Tue Jan 24 09:59:42 2006

Method C:\MSDCHEM\1\METHODS\TCLP2.M renamed as C:\MSDCHEM\1\METHODS\TCLP.M
Sat Mar 04 09:28:42 2006

Method C:\MSDCHEM\1\METHODS\TCLP2.M renamed as C:\MSDCHEM\1\METHODS\TCLP.M
Wed Oct 17 08:41:05 2007

Method C:\MSDCHEM\1\METHODS\TCLP.M renamed as C:\MSDCHEM\1\METHODS\PYONLY.M
Thu Jan 24 16:10:28 2008

Method C:\MSDCHEM\1\METHODS\TCLP.M renamed as C:\MSDCHEM\1\METHODS\TCLP8270.M
Mon Aug 18 09:12:14 2008

Method C:\MSDCHEM\1\METHODS\TCLP.M renamed as C:\MSDCHEM\1\METHODS\3.M
Fri Jan 02 10:08:15 2009

Method C:\MSDCHEM\1\METHODS\TCLP.M renamed as C:\MSDCHEM\1\METHODS\0.M
Thu Jan 08 15:36:34 2009

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[redacted] Feb 11 11:21:16 2009

Method C:\MSDCHEM\1\METHODS\TCLP.M renamed as C:\MSDCHEM\1\METHODS\TCLP3239.M

BECKTON ENVIRONMENTAL LABORATORIES, INC.

HISTORY OF REVISIONS

SOP NUMBER: _____

REVISION NO.	EFFECTIVE DATE	PAGES REVISED	REASON FOR CHANGE	APPROVED BY: